Europäisches Patentamt
European Patent Office
Office européen des brevets

European Patent Office
80298 MUNICH
GERMANY
Tel. +49 (0)89 2399 - 0
Fax +49 (0)89 2399 - 4465

Henkel, Feiler & Hänzel
Patentanwälte
Maximiliansplatz 21
80333 München
ALLEMAGNE

EINGEGANGEN
17. März 2011
Henkel, Feiler & Hänzel

Formalities Officer
Name: Durany Rivera, Angela
Tel.: 8114
or call:
+31 (0)70 340 45 00

Date: 17-03-2011

Reference	Application No./Patent No.
10613/CH/ajk	98941672.2 - 2111 / 1033347
Applicant/Proprietor: ISHIHARA SANGYO KAISHA, LTD., et al	

BRIEF COMMUNICATION

Subject:
- ☐ Your letter of
- ☐ Our telephone conversation of
- ☐ Communication of
- ☑ Oral P. 13-04-2011

Enclosure(s):
- ☐ Letter from the proprietor of the patent of
- ☑ Letter from the opponent of 11-03-2011 (two letters with enclosures)
- ☐ Copy (copies)

Communication:

☐ A letter from the opponent /proprietor was received on ..... . The documents specified as patent documents in this letter are now available via the Register Plus online service under **http://www.epoline.org** (see Special edition No. 3, OJ EPO 2007, J.2). Should you wish to receive these documents in paper format, they will be supplied to you free of charge if specifically requested on receipt of this communication (OJ EPO 2009, 434).

☐

For the Opposition Division

Europäisches Patentamt • European Patent Office • Office européen des brevets

☐ **Registered letter**

TER MEER STEINMEISTER & PARTNER GbR · PATENTANWÄLTE
EUROPEAN PATENT ATTORNEYS

Dipl.-Phys. Peter Urner
Dipl.-Ing. Gebhard Merkle
Dipl.-Phys. Bernhard P. Wagner
Dipl.-Chem. Dr. Christian Hollatz
Dipl.-Ing. Jörg Riemann
Dipl.-Chem. Dr. Bernd Aechter
Dipl.-Phys. Dr. Matthias Brittinger
Manfred Wiebusch*

Consultants:
Dipl.-Chem. Dr. Nicolaus ter Meer
Dipl.-Ing. Helmut Steinmeister*

Mauerkircherstrasse 45
D-81679 MÜNCHEN

Telefon: (089) 98 44 37
Telefax: (089) 98 79 52
Telefax: (089) 98 27 199
www.termeer.de
VAT Reg. No: DE 130 747 219
St.-Nr./ Tax No: 641/17309

ter Meer Steinmeister & Partner · Postfach 86 07 69 · D-81634 München

Europäisches Patentamt
Erhardtstraße 27
80469 München

EPO - Munich
57
11. März 2011

URGENT!

11.03.2011
Be/vb

Opposition against European Patent EP 1 033 347 B1
European Patent Application No. 98 941 672.2
Title: Hollow fine powder, flaky fine titanium oxide powder prepared by pulverizing said hollow fine powder, and process for preparing the both
Proprietors: Ishihara Sangyo Kaisha Ltd. et al.
Opponent: Dr. Nicolaus ter Meer
Case: OPJH-013EP

With separate submission of today, document D7, the English abstract of JP 05-229900 is being submitted. Since the Opponent wishes to rely on the abstract only, provision of a translation of the full document was not deemed necessary. Nevertheless, as a precautionary measure, a computer translation of the entire document D7 as well as the Japanese original are filed herewith.

Should the Opposition Division consider it necessary that a "proper" translation be filed, Opponent would be obliged if the Opposition Division could inform the Opponent accordingly so that it may be filed in time for all involved to consider.

Respectfully submitted for the Opponent.

Dr. Christian Hollatz

Enclosure:
D7 – JP 05-229900 and English machine translation

* Büro Bielefeld: Artur-Ladebeck-Str. 51, D-33617 BIELEFELD
Telefon: (0521) 914 940 Telefax: (0521) 914 94 20

TER MEER STEINMEISTER & PARTNER GbR · PATENTANWÄLTE

EUROPEAN PATENT ATTORNEYS

Dipl.-Phys. Peter Urner
Dipl.-Ing. Gebhard Merkle
Dipl.-Phys. Bernhard P. Wagner
Dipl.-Chem. Dr. Christian Hollatz
Dipl.-Ing. Jörg Riemann
Dipl.-Chem. Dr. Bernd Aechter
Dipl.-Phys. Dr. Matthias Brittinger
Manfred Wiebusch*

Consultants:
Dipl.-Chem. Dr. Nicolaus ter Meer
Dipl.-Ing. Helmut Steinmeister*

Mauerkircherstrasse 45
D-81679 MÜNCHEN

Telefon: (089) 98 44 37
Telefax: (089) 98 79 52
Telefax: (089) 98 27 199
www.termeer.de
VAT Reg. No: DE 130 747 219
St.-Nr./ Tax No: 641/17309

ter Meer Steinmeister & Partner · Postfach 86 07 69 · D-81634 München

Europäisches Patentamt
Erhardtstraße 27
80469 München

EPO - Munich
57
11 März 2011

11.03.2011
Be/vb

Opposition against European Patent EP 1 033 347 B1
European Patent Application No. 98 941 672.2
Title: Hollow fine powder, flaky fine titanium oxide powder prepared by pulverizing said hollow fine powder, and process for preparing the both
Proprietors: Ishihara Sangyo Kaisha Ltd. et al.
Opponent: Dr. Nicolaus ter Meer
Case: OPJH-013EP

In response to the Summons to attend Oral Proceedings pursuant to Rule 115(1) EPC dated November 18, 2010.

In addition to the arguments provided in the writ of opposition, the following is submitted.

1. New document

In response to the opposition, the Patent Proprietor amended claim 13 as granted by incorporating features taken from the description. In response thereto, an additional document is submitted herewith:

D7: JP 05-229900 (English abstract)

2. Lack of Novelty (Art. 100(a) and Art. 54 EPC)

The original attacks on novelty of the various claims are maintained, with the exception of claim 13, and briefly supplemented as indicated below.

* Büro Bielefeld: Artur-Ladebeck-Str. 51, D-33617 BIELEFELD
Telefon: (0521) 914 940 Telefax: (0521) 914 94 20

2.1 Claim 1

Regarding claim 1, the Proprietor argued that in the reply dated October 17, 2008 that „Document D4 discloses in Example 2.1 (see the paragraph bridging pages 7 and 8 of the document D4) a spray drying process wherein titanium tetrachloride is used as a raw material. In contrast, according to the present invention an exfoliated titanium sol is used as starting material which may be subjected to a spray drying step." (page 6, lines 14-19 of the reply).

However, in Examples 1.1 to 1.12 of the document D4, microballoons were obtained using the dispersions of metal oxides such as manganese oxide and other metal oxides listed in Table 1. Further, in document D4, "titanium oxide" is listed as an oxide dispersed in a liquid medium (page 2, line 48). Furthermore, document D4 discloses that it is possible to produce crystalline micoballoons made of an oxide of a transition metal such as titanium (page 4, line 51-52). Therefore, the disclosure of document D4 is not limited to the disclosure of the one example, as the Proprietor seems to suggest.

In light thereof, the Proprietor's conclusion cannot be followed.

2.2 Claim 7

The question of overlap of the subject-matter of claim 7 and the disclosure of document D3 involves an assessment of accuracy. The Opposition Division pointed to the patent indicating only one decimal place in an example.

However, as pointed out before, even if the accuracy of the variable in question could be given to two decimal places, the lower limit of 0.57 defined in claim 7 still encompasses values of 0.565 and the upper limit of 0.75 in document D3 still encompasses values of 0,754, as mentioned before. This means that claim 7 covers compounds down to and including $Ti_{1.812}O_4^{-0.754}$ whilst document D3 discloses compounds up to $Ti_{1.812}O_4^{-0.753}$. Thus, the ranges clearly overlap, based on the universally applicable rules of rounding figures.

Claim 7 is therefore regarded as lacking novelty over document D3.

2.3 Claim 15

With regard to claim 15, it shall merely be noted herein that the Patent Proprietor's argument defending novelty relies on features not recited in the claim. It is therefore fully concurred with the Opposition Division's finding of lack of novelty.

3. Lack of Inventive Step

3.1 Amended claim 13

Document D1 discloses, amongst others, compounds of the formula $K_{0.8}(Li_{0.266}Ti_{1.733})O_4$.

In light thereof, granted claim 13 has been amended such that the mixed alkali metals are now limited to a combination of Rb or Cs rather than K with Li. The difference to the disclosure of D1 therefore resides in the alkali metal ion. For an assessment of a potential technical effect of such a difference, it is referred to the examples of the patent, which are summarized in a list in Annex I submitted herewith for ease of illustration. As evident from the table, the mixed alkali metal titanate containing Cs corresponding to new claim 13 is prepared in Example 2, K containing alkali metal titanates no longer covered by claim 13 are prepared in examples 1 and 3.

For the preparation of a layered titanic acid compound, the K containing titanate of example 1 is used in example 4, the Cs containing titantate is used in example 5 and the K containing titanate of example 3 is used in example 6. For the synthesis of exfoliated titania sol, the layered titanic acid compound of example 4 is used in example 7, the layered titanic acid compound of example 5 is used in example 8,and the layered titanic acid compound of example 6 is used in example 9.

However, only the exfoliated titania sol of example 7 was used in examples 10 and 11, while the exfoliated titania sol of example 8 was not used in any examples for the preparation of fine follow powder. No advantage of the Cs containing titanate is therefore apparent compared to the K containing titanate. In light thereof, the problem to be solved can only be seen in the provision of a further mixed alkali metal titanate.

Replacing K in $K_{0.8}(Li_{0.266}Ti_{1.733})O_4$ as disclosed in D1 with Rb or Cs would have been obvious to the skilled person. Document D1 discloses K, Rb, Tl and Cs as possible ions of insertion (first page, left column, lines 10/11 from top of first paragraph). It further teaches compounds wherein the alkali metals are simply exchanged for one another, such as $K_{0.8}Mg_{0.4}Ti_{1.60}O_4$ vs. $Rb_{0.8}Mg_{0.4}Ti_{1.60}O_4$ (Table 1) and $Rb_xMg_yTi_{2-y}O_4$ vs. $K_xMg_yTi_{2-y}O_4$ (legend of Figure 3). Therefore, the person skilled in the art striving to provide further mixed alkali metal titanates would have been prompted to replace K with Rb or Cs. The subject-matter of claim 13 is therefore not based on an inventive step.

The subject-matter of claim 13 would also be obvious from a combination of documents D1 and D7. D7 discloses a rhombic layered plate crystal expressed by the general formula $A_xM_yTi_{2-y}O_4$, wherein A is K, Rb or Cs, M is Mg^{2+}, Ni^{2+}, Zn^{2+}, Cu^{2+}, Fe^{3+}, Al^{3+}, Ga^{3+} or Mn^{3+}, x and y are in a range of 0.5 to 1.0 and of 0.25 to 1.0. In analogy to document D1, document D7 therefore

teaches that K may be replaced with Rb or Cs. The skilled person striving to provide a further mixed alkali metal titanate would therefore be directly led to the claimed solution.

3.2 Claim 1

The Patent Proprietor argued that a fine hollow powder as claimed in claim 1, which is obtained using a specific reactive mixed alkali metal titanate as claimed in new claim 13 as a starting material for the preparation of a layered titanium acid compound according to claim 15 and an exfoliated titantia sol according to claim 7, showed higher absorbability and better dispersibility in liquid media. However, this argument cannot be followed.

The description of the patent discloses only test examples 1 and 2. In test example 1, sample L (which is the fine hollow powder according to example 11, which in turn is based on the K containing titanate of example 7), sample N (which is the thin flaky titanium oxide powder obtained by pulverizing the fine hollow powder of example 11 and is therefore equally based on the K containing titanate of example 7) and sample O (which is the planar flaky titanium oxide powder obtained by drying, heat-treating and pulverizing the exfoliated titania sol of example 7) are evaluated.

Therefore, all evaluated examples are prepared from the K containing titania sol of example 7. Accordingly, there is no data in the patent that would allow a comparison to the prior art.

3.3 Claims 7 and 15

With respect to the exfoliate titania sol according to claim 7, the following is noted with respect to any conclusions that may be drawn from the examples of the patent, should novelty of claim 7 unexpectedly be accepted:

In comparative example 1, exfoliated titania sol is prepared according to the process of prior art document D3. However, the exfoliated titania sol thus prepared is not specifically evaluated in the description. The description discloses in paragraph [0096] that "No solid precipitate was observed in Sample J, even after being left standing for a while, but it was found at the time of shaking after one day as well as after two days that solid matters were precipitated by leaving it standing for a while, showing that the dispersion was not satisfactory."

With respect to the exfoliated titania sol of example 7 (according to the patent), the description of the patent discloses in paragraphs [0083] and [0084] that "No solid precipitate was observed in Sample G even after being left standing for a while. Sample G was separated into a substan-

tially clear supernatant liquid and a paste-like precipitate by centrifugation at 15,000 rpm for 30 minutes. The precipitate was presumed to be aggregates of particles dispersed in the sol."

This would seem to suggest that there is a difference between example 7 and comparative example 1.

However, attention is drawn to the disclosure in paragraph [0017] of document D3. It states that "The obtained titania sol was milky white, and even when the sol is left for a long time without treatment, no solid is observed on the flask bottom" and further "When the suspension of the invention was centrifuged at 15,000 rpm for about 30 minutes, a pasty solid was deposited on the centrifuge bottom. Since the supernatant liquid was almost transparent, the deposit is considered to be an aggregate of fine particles suspended in the sol." with respect to the titania sol obtained by the process according to document D3.

In light of the above, it becomes apparent that there is no difference in dispersibility in liquid media between comparative example 1, which corresponds to prior art document D3, and example 7 of the patent.

In the absence of any surprising technical effect, the subject-matter of claim 7 cannot be regarded as being based on an inventive step.

The same would apply *mutatis mutandis* to claim 15, for which no technical effect can be derived from the patent, either.

In addition, document D7 discloses that rhombic layered titanic acid plate crystals represented by the general formula $H_xM_yTi_{2-y}O_4 \cdot n\ H_2O$ (wherein M is Mg^{2+}, Ni^{2+}, Zn^{2+}, Cu^{2+}, Fe^{3+}, Al^{3+}, Ga^{3+} or Mn^{3+}, x and y are in a range of 0.5 to 1.0 and of 0.25 to 1.0, $n \leq 2$) is obtained by treating a rhombic layered plate crystal expressed by a general formula $A_xM_yTi_{2-y}O_4$ (wherein A is K, Rb or Cs, M is Mg^{2+}, Ni^{2+}, Zn^{2+}, Cu^{2+}, Fe^{3+}, Al^{3+}, Ga^{3+} or Mn^{3+}, x and y are in a range of 0.5 to 1.0 and of 0.25 to 1.0) with an acidic aqueous solution so as to exchange the A ions coordinated between the layers with H^+ ion or H_3O^+ ions.

Accordingly, it would have been obvious for a person skilled in the art to treat $K_{0.8}(Li_{0.266}Ti_{1.733})O_4$ as disclosed in D1 with an acidic aqueous solution so as to obtain a layered titanic acid compound as claimed in claim 15. Furthermore, it would have also been obvious for a person skilled in the art to treat the obtained layered titanic acid compound with a basic compound so as to obtain an exfoliated titania sol as claimed in claim 7, as taught by document D3.

Respectfully submitted for the opponent.

Dr. Christian Hollatz

Enclosures:
D7
Annex 1 - tabular overview of examples of EP 1 033 347 B1

PATENT ABSTRACTS OF JAPAN

(11)Publication number : **05-229900**
(43)Date of publication of application : **07.09.1993**

(51)Int.Cl. **C30B 29/32**
C01G 23/00
C30B 29/64

(21)Application number : **04-070206**
(22)Date of filing : **20.02.1992**

(71)Applicant : **NATL INST FOR RES IN INORG MATER**
(72)Inventor : **FUJIKI YOSHINORI**
SASAKI TAKAYOSHI

(54) PRODUCTION OF PHOMBIC LAYERED TITANIC ACID PLATE CRYSTAL REPRESENTED BY HXMYTI2-YO4-NH2O

(57)Abstract:
PURPOSE: To produce new rhombic layered titanic acid plate crystal represented by HxMyTi2-yO4.nH2O.
CONSTITUTION: The objective rhombic layered titanic acid plate crystal represented by a general formula HxMyTi2-yO4.nH2O (Provided that M shows Mg2+, Ni2+, Zn2+, Cu2+, Fe3+, Al3+, Ga3+ or Mn3+, (x) and (y) are respectively within a range of 0.5 to 1.0 and of 0.25 to 1.0. (n) is ≤2) is obtained by treating a rhombic layered plate crystal expressed by a general formula AxMyTi2-yO4 (Provided that A shows K, Rb or Cs. M is Mg2+, Ni2+, Zn2+, Cu2+, Fe3+, Al3+, Ga3+ or Mn3+. (x) and (y) are respectively within a range of 0.5 to 1.0 and of 0.25 to 1.0) with an acid aqueous solution so as to exchange A ion coordinated between the layers with H+ ion or H3O+ ion. As the aqueous acid solution for extracting A ion by acid treatment, any aqueous acid solution may be used and an aqueous hydrochloric acid solution is most suitable. The plate crystal is useful as a cation exchanger, a filter, a proton conductor and a catalyst.

!

Annex 1 – Examples in the description of EP 1 033 347 B1

<table>
<tr><td>Synthesis of mixed alkali metal titanate</td><td>Ex. 1
Ex. 2
Ex. 3</td><td>$K_{0.8}$ $[Li_{0.266}Ti_{1.733}]$ O_4
$Cs_{0.7}$ $[Li_{0.233}Ti_{1.766}]$ O_4
$K_{0.8}$ $[Li_{0.266}Ti_{1.733}]$ O_4</td></tr>
<tr><td>Synthesis of layered titanic acid compound</td><td>Ex. 4
Ex. 5
Ex. 6</td><td>Synthesis using $K_{0.8}$ $[Li_{0.266}Ti_{1.733}]$ O_4 obtained in Ex.1
Synthesis using $Cs_{0.7}$ $[Li_{0.233}Ti_{1.766}]$ O_4 obtained in Ex.2
Synthesis using $K_{0.8}$ $[Li_{0.266}Ti_{1.733}]$ O_4 obtained in Ex.3</td></tr>
<tr><td rowspan="2">Synthesis of Exfoliated titania sol</td><td>Ex. 7
Ex. 8
Ex. 9</td><td>Synthesis using layered titanic acid compound obtained in Ex. 4
Synthesis using layered titanic acid compound obtained in Ex. 5
Synthesis using layered titanic acid compound obtained in Ex. 6</td></tr>
<tr><td>Comp. Ex.1</td><td>Synthesis using $Cs_xLi_{2-x/4}O_4$</td></tr>
<tr><td>Synthesis of fine hollow powder</td><td>Ex. 10
Ex. 11</td><td>Synthesis using exfoliated titania sol obtained in Ex. 7
Dring at 200°C
Synthesis using exfoliated titania sol obtained in Ex. 7
Heat treatment at 670°C (Sample L)</td></tr>
<tr><td>Preparation of thin flaky titanium oxide powder</td><td>Ex. 12
Ex. 13</td><td>Pulverizing fine hollow powder obtained in Ex.10
Pulverizing fine hollow powder obtained in Ex.11
(Sample N)</td></tr>
<tr><td>Preparation of planar titanium oxide powder</td><td>Comp. Ex. 2</td><td>Pulverizing exfoliated titania sol obtained in Ex.7 after heat treatment at 650°C for one hour
(Sample O)</td></tr>
<tr><td>Evaluation Test</td><td>Test Ex. 1</td><td>Comparison of Samples L, N and O
<table>
<tr><th></th><th>Cosmetics 1 (Sample L)</th><th>Cosmetics n (Sample N)</th><th>Cosmetics o (Sample O)</th></tr>
<tr><td>Extendability on skin</td><td>10.0</td><td>10.0</td><td>2.3</td></tr>
<tr><td>Sense of clearness on skin</td><td>7.6</td><td>8.3</td><td>2.1</td></tr>
</table></td></tr>
<tr><td></td><td>Test Ex. 2</td><td>Comparison of Sample L and silica glass beads
Sample L : Fig. 11 and Fig. 12
Silica glass beads : Fig. 13 and Fig. 14</td></tr>
</table>